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14. ABSTRACT
Improving materials processing capabilities is of fundamental importance to enable DOD and the Air Force to meet their future materials requirements for advanced applications. However, processing problems are increasingly more difficult as we seek to manufacture new materials with greater control over material microstructure, meet more stringent performance requirements while significantly reducing cost and time to market. To meet these challenges, we have been developing a controls based approach utilizing real-time sensors. The DURIP grant provided funds to implement three advanced materials process control applications: crystal growth for advanced opto-electronic semiconductors for high-bandwidth communications and detection, plasma deposition for protective coatings critical for engines, turbines, and space propulsion systems, and CVD, an enabling technology for many critical applications in aerospace, engines, manufacturing, and micro and opto-electronics. An overview of the equipment acquired and research results is provided.

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Real-Time Control for Advanced Materials Processing Applications

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Abstract

Improving materials processing capabilities is of fundamental importance to enable DOD and the Air Force to meet their future materials requirements for advanced applications. However, processing problems are increasingly more difficult as we seek to manufacture new materials, achieve greater control over material microstructure, meet more stringent performance requirements while significantly reducing cost and time to market. To meet these challenges, we have been developing a controls based approach utilizing real-time sensors. The DURIP grant provided funds to implement three advanced materials process control applications: in crystal growth for advanced opto-electronic semiconductors for high-bandwidth communications and detection, in plasma deposition for protective coatings critical for engines, turbines, and space propulsion systems, and in CVD, an enabling technology for many critical applications in aerospace, engines, manufacturing, and micro and opto-electronics.

Contents:

I Introduction

Improving materials processing capabilities is of fundamental importance to enable DOD and the Air Force to meet their future materials requirements in advanced applications. However, processing problems are increasingly more difficult as we seek to manufacture new materials with achieve greater control over material microstructure, meet more stringent performance requirements while significantly reducing cost and time to market. To meet these challenges, we have been developing a controls based approach utilizing real-time sensors. The DURIP grant provided funds to implement three advanced materials process control applications: in crystal growth for advanced opto-electronic semiconductors for high-bandwidth communications and detection, in plasma deposition for protective coatings critical for engines, turbines, and space propulsion systems, and in CVD, an enabling technology for many critical applications in aerospace, engines, manufacturing, and micro and opto-electronics.

The equipment obtained through this grant enables advancing both fundamental knowledge-base of these processes as well as demonstrating the advanced control concepts, speeding adaptation of the results. Beyond the specific projects, the research supported by this grant furthers the development of a controls based approach to materials processing and provides a formative experience for graduate and undergraduate students who participate in this research. This approach is based on an integrated effort of physical modeling, sensor development, system design, and control development.

The equipment supports on-going, funded research projects including a) bulk Czochralski crystal growth for opto-electronic materials (funded by ARPA MURI program), b) plasma deposition for spray coatings including advanced engines and space applications (NSF and INEEL), and c) CVD for both opto-electronic applications such as multi-layered coatings, as well as protective coatings for engines and cutting tools. To insure that we are focused on problems critical to DOD/Air Force and industry as well as speed technology transfer, we have developed a series of industrial and Air Force Laboratory collaborations (see section VII).

These processes are related in that they are all involve control of thermal-fluid systems and it is necessary to consider the multi-variable nature of the process in order to achieve the desired materials objectives. The also share similar sensing capabilities that we are currently implementing. Equipment obtained through this grant serves as a test-bed to develop and demonstrate these advanced sensors and control systems. As such, the equipment has been selected that is near production scale in order to speed technology transfer.

II Project and Research Summary

DURIP project funds were used to establish three state-of-the-art facilities for developing and demonstration of advanced materials process control applications in the areas of a) crystal growth, b) chemical vapor deposition, and c) plasma deposition.

In CVD, funds were used to provide advanced data acquisition and sensing capabilities for a 3 hot-zone hot-walled reactor. A mass spectrometer has been added that enables real-time sensing of reaction chemistry that is important both for validating control models that have been developed and implementation of real-time control. Results include validation of detailed partial pressure dynamics(sec.VI.4) which are a foundation for the advanced control design.

In crystal growth, funds were used to obtain a industrial scale puller that is being used to implement advanced control concepts and obtain insight into realistic system characteristics which vary both machine-to-machine and system-to-system. Important experimental results to-date(sec.VI.3) include obtaining actuator data sets for various growth conditions, which provide both the basis for validating numerical process-equipment models, as well as revealing critical operating features that serve as the basis for developing advanced control concepts including the need for gain scheduling, and feedforward disturbance control.

In plasma deposition, we have developed an experimental commercial scale plasma spray facility that enables both characterization studies as well as implementation of real-time control. Research results (sec.VI.2) include system characterization which revealed the performance limitations posed by the current open-loop practices. In addition, the system was characterized in order to develop appropriate control structures and implemented closed loop control has been demonstrated.

III Budget Summary

Equipment purchased under this grant had a total value of $173,578. Of this, $120,000 came from DURIP, $30,000 (17%) came from direct University matching funds, $17,189 (10%) came from extended discounts and gifts from Industrial partners/equipment vendors, and $6,389 (4%) came from other research grants funds. Comparing only the direct DURIP and University matching funds, the support provided was 80% and 20% respectively.

IV Instrumentation Acquired

IV.1 Czochralski Crystal Puller

We have established collaborative working relationships with two important equipment manufactures: we are working with Kayex, one of the major American equipment manufacturers (Kayex is the only domestic company that was awarded a contract by MEMC for supplying a 300 mm Si puller).

Since Kayex no longer manufacture equipment of suitable size for research level demonstration of closed loop control (current commercial scale systems for Silicon are

designed for growing very long boule of 150-200 mm crystals), we have also established a relationship with GTI equipment, which is a rapidly growing company in the area of specialty pullers and equipment. They have refurbished a Varian puller , upgrading key systems including the vacuum chamber and power supply.

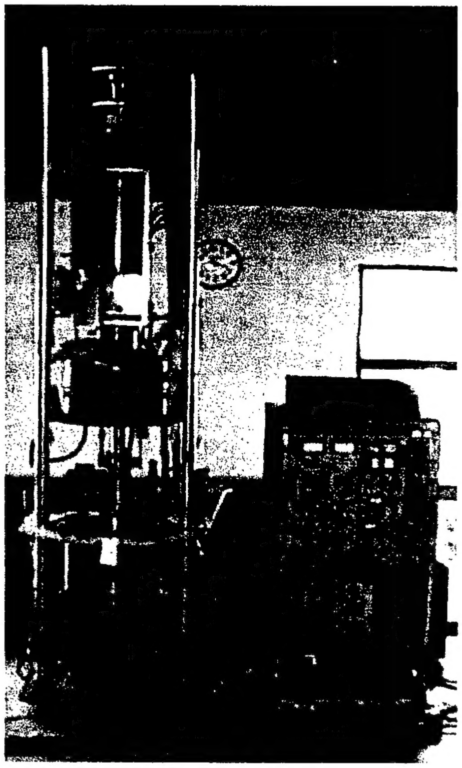

Fig. 1: Refurbished Varian puller, power supply, and control console

For our research, we sought a system large enough to capture the critical scale issues without requiring the resources of production scale equipment. The puller is designed to utilize either a 6 or 8 inch crucible. The 8 inch crucible can handle a 8 kg melt. The heater is a graphite resistance type powered by a 75 kW power supply and can be controlled to maintain a desired heater temperature measured by a thermocouple in close proximity to the heater. Basic actuator requirements include independent drives for both seed rod and crucible (seed rod should have a variable pull speed of 0-20 inches per hour, rotation in either direction (0-50 rpm), and a total traverse of 36-60 inches). Crucible actuators include lift (0-10 inches per hour) and rotation in either direction (0-25 rpm). In addition, we are also utilizing a puller was donated to us from MEMC.

IV.2 CVD

Currently, no closed loop control is used with respect to the ultimate objectives of CVD, ie the quality of the coating. Our research has been focused on pushing control closer to this ultimate objectives, and we have used the DURIP grant to obtain major instrumentation for use in our custom designed CVD reactor system.

We have developed an experimental CVD hot wall reactor that is computer controlled utilizing the National Instruments data acquisition and control cards obtained with the DURIP grant. The system actuators include a 3 zone resistance heater (with local Eurotherm controllers), MKS gas mass flow controllers (N_2, H_2, Ar, He), Tylan vapor mass flow controller for T_iCl_4, and a MKS pressure control system based on N_2 bleed throttle. Sensors include pressure transducer, thermocouples for temperature distribution, and a microbalance used to measure growth rate.

Funds from the grant were also used to obtain a MKS mass spectrometer in order to measure in real-time the concentration variation within the rector in order to gain insight into process chemistry, verify model equations, and implement real-time feedback. Since our CVD applications operate at between 10 torr and 1 atm, a capillary inlet system is required, along with turbo pump. A differential pressure quadruple RGA system is used (DPS-C200-1T) for heavy ions.

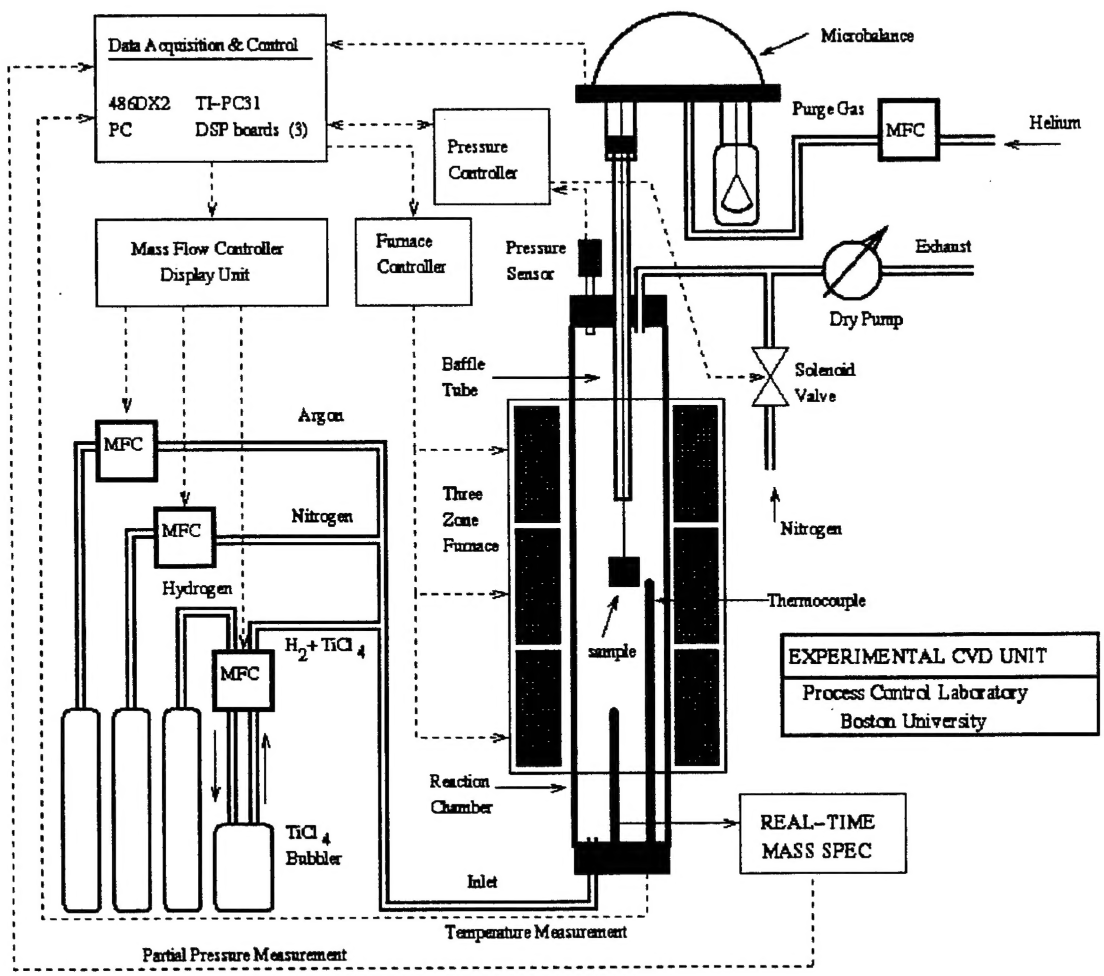

Fig 2: Schematic of closed-loop CVD control system

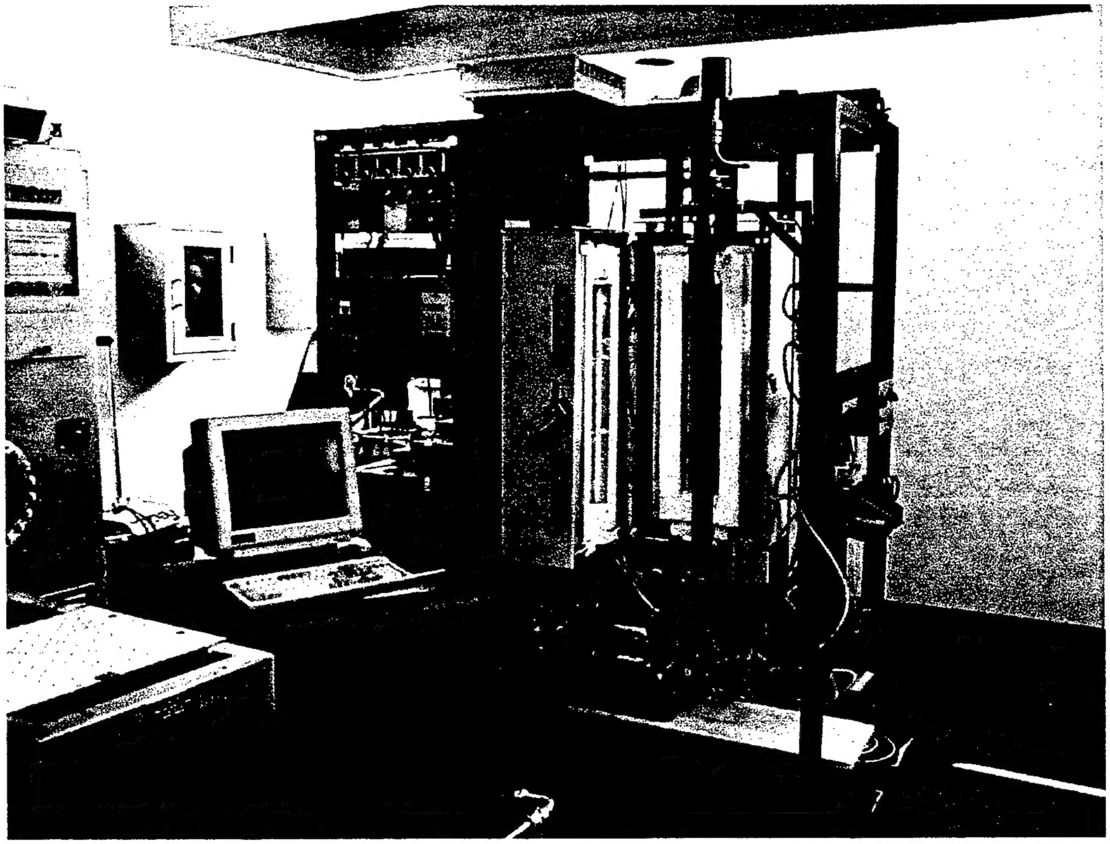

Fig 3: CVD reactor and control equipment.

IV.3 Data Acquisition

Our strategy is to customize data acquisition and control by utilizing well established software and hardware capabilities from National Instruments. Data acquisition is used the AT-MIO-64E-3 board that has 64 single and 32 differential inputs, 500 K sampling rate, 12 bit resolution, with 2 analog output channels and 8 digital outputs. Output will be from the AT-A10 board that has 10 12 bit analog output channels and 8 digital outputs. These boards are installed in an industrial computer with expanded back plane.

IV.4 Plasma Deposition

In order to utilize industry-scale equipment recognized by the plasma spray community, we decided to procure equipment from Praxair. Praxair is an American company that leads in equipment manufacturing of thermal spray systems, development of new coatings for clients, and is also has a large coating business. We met with Dr. Daryl Crawmer, Director of Engineering at Praxair, Thermal Spray Products, to discuss their interest in developing advanced control systems for plasma spray.

Equipment needed to develop a basic cell for demonstration includes powder feeder, power supplies and plasma gun, spray chamber, high frequency power supply to initiate the arc. In order to customize the advanced control system, we have decided to develop our own control console, coordinating mass flow controllers, main power set point, and recording real-time data acquisition including cooling water, plasma/particle measurements, and substrate temperature. Features of the system include:

Model SG100 plasma spray gun: is a multi-mode plasma spray gun designed for a broad range of thermal spray applications. It can be used both in high volume production environments requiring high quality, rapid, and uniform repeatable coatings, as well as lower volume applications. The unique design of the gun permits both internal and external powder injection capability.

Model PS 100 plasma power source is a 100 kW constant current power supply designed for plasma spraying. Advanced solid-state control circuitry and high power design provide precise control and optimum stability for plasma spraying. Setpoint parameters are entered via the controller which can be either held at a set point or changes during operation.

V Budget Breakdown

Application	Equipment	DURIP Funds	BU Match Funds	Industrial Funds/Discounts	Other Grants	Total	
CVD							
	Mass Spec	29,852		7,463		37,315	
	Misc. CVD equip.	2,384				2,384	
						39,699	23%
Plasma Deposition	PS-100 Power Supply	13,440		707		14,147	
	High Freq. Power Supply	2,280		120		2,400	
	SG 100 Gun	3,520		185		3,705	
	Powder feeder			600	6,180	6,780	
	Control Interface		1,843	114	209	2,166	
	Plasma Enclosure	9,746	2,152			11,898	
	Installation		5,000			5,000	
	Dust Collector		18,655			18,655	
						64,751	37%
Plasma Sensor							
	HP Digital Oscill.	5,990				5,990	
	Optical Detectors	2,180				2,180	
	Fiber	2,000				2,000	
	Optical Components	534				534	
						10,704	6%
Crystal Growth	Varian Puller	34,174		5,000		39,174	
	rigger		2,350			2,350	
	vacuum system			3,000		3,000	
						44,524	26%
Data Acq. & Control	PC (Dell)	2,220				2,220	
	Sun	7,633				7,633	
	National Instruments	4,047				4,047	
						13,900	8%
Totals		120000	30000	17189	6389	173,578	
	overall %	69%	17%	10%	4%		
	DURIP/BU %	80%	20%				

VI Research Results

VI.1 Educational Impact

This grant has supports of the development of a systems based approach to materials processing. Control and system theory provide a useful framework to address important issues of materials processes including control structure development, system design, and operating regime determination. Such a systems based approach complements numerical modeling, instrumentation development, and experimentation by providing a new perspective on how to approach materials processing problems. Such an approach is being adapted into the university curriculum for materials and manufacturing engineers.

At Boston University, we have developed a new course, MN 507 Process Modeling and Control, which is offered to both seniors and graduate students, both for our on-campus program, and in a special Executive formatted program for Manufacturing Engineers from industry. Results from the experimental systems funded by this grant are used throughout the course, in terms of special problems, case studies, and has been incorporated in written articles used in teaching the course.(Gevelber,1999)

Developing and implementing the experimental facilities for crystal growth, CVD, and plasma spray has provided an important educational experience for our students. Besides the direct experience of combining theory and practice, they have been exposed to issues related to the reduction of theory to practice, essential for those who are to be leaders in keeping American industry at the forefront of manufacturing.

Both graduate and undergraduate students were given the opportunity to obtain hands-on experience through work on design and implementing the experimental hardware funded under this grant, as well as conducting experiments. **Of particular note, undergraduate Joseph Owen was awarded a Barry Goldwater Scholarship for his work.**

Graduate Students	Undergraduates
Danielle Wilson (Crystal) Ning Duanmu (Crystal)	Clinton Reed (Crystal)
Sanjeev Mathur (CVD)	Joseph Owen (CVD) Michael Murphy (CVD) Erin Martell (CVD) Nathan Spiker (CVD) Scott Kreamer (CVD)
Rajesh Kahre (Plasma)	Sarah Felix (Plasma) Jorge Champlin (Plasma) Luke Thulin (Plasma) Doug DiSabello (Plasma)

VI.2 Control of Plasma Deposition

Plasma spray has been an enabling technology in many applications. However, the plasma-spray process features complex plasma-particle interactions with significant variations and distributions, which limit the process potential due to the significant variations in the particle state that occur run-to-run and during a long deposition run for a large part, the limited the ability to maintain a narrow operating window, precluding applications requiring tight control of resulting coating structure, and the complexity in developing new process recipes to achieve specific set of coating materials objectives.

Currently, plasma spray deposition is operated for the most part in an open-loop fashion, in that actuator set points (such as for current and flow rates) are developed empirically based on the user's process knowledge/experiments. Thus, there is no automatic adjustment of input levels to maintain the process/particle state in spite of process variations nor easy-to-use method to determine the required set-points to achieve a desired set of coating properties. Process variations that occur include electrode wear over the 40-50 hour life, operation that entails multiple on-off cycles, and after maintenance. Closed loop control, in contrast, offers the opportunity to compensate for such variations as well directly achieve the required conditions to achieve a desired coating structure.

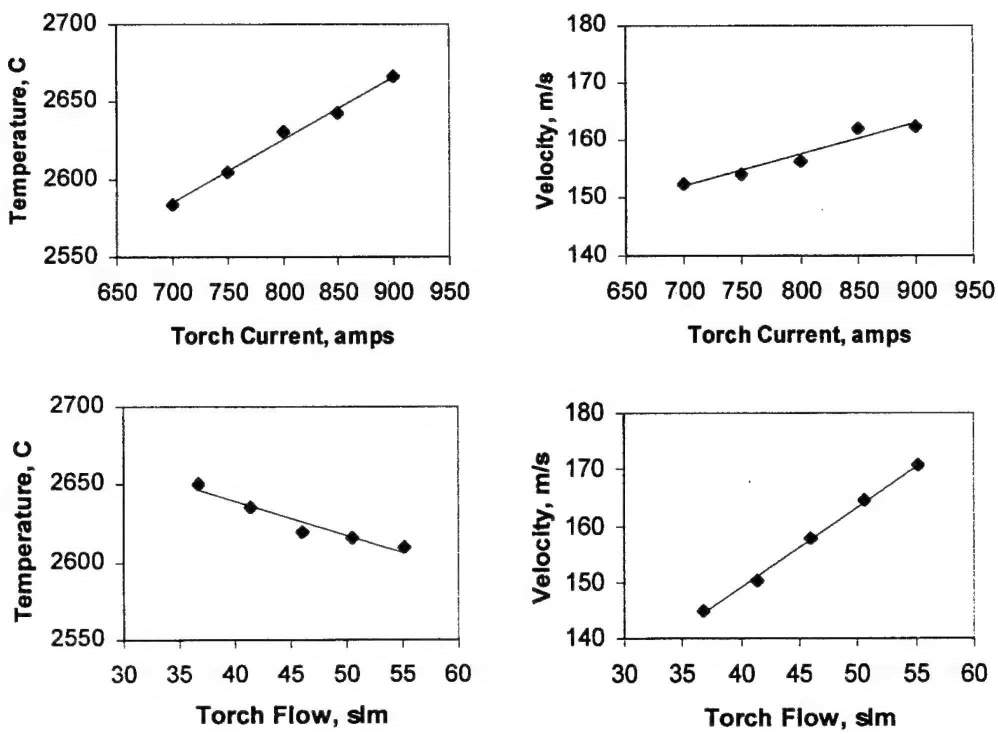

Figure 4: Steady-state input-output experimental results

While there has been significant experimental and modeling work (e.g., Vardelle, 1994, Pfender, 1991, Sampath, 1996) reported on the relationship between particle state (temperature/velocity), the deposition process (splat formation, solidification dynamics), and the resulting coating structure, there is little reported on the aspects needed to develop an appropriate real-time control strategy and the relationship between spray conditions and resulting coating structure. This has been the aim of our experiment and modeling research.

Our initial steady-state input-output study was conducted by varying each input while measuring the three outputs. Inputs considered were the total plasma gas flow rate, torch current, and carrier gas flow rate, while outputs were the averaged particle temperature and velocity, as well as the centroid position(a total 9 curves). Four representative relations are shown in Fig 4 (starting at the lower end of the range for that variable and sweeping sequentially to the higher end).

Eight of the input/output curves have dominant linear relationships (except for temperature-carrier gas flow rate relation). A least squares linear fit to the data yields the transfer matrix (Fig.5) which is scaled by allowable perturbations of the inputs and sensitivity values for the outputs.

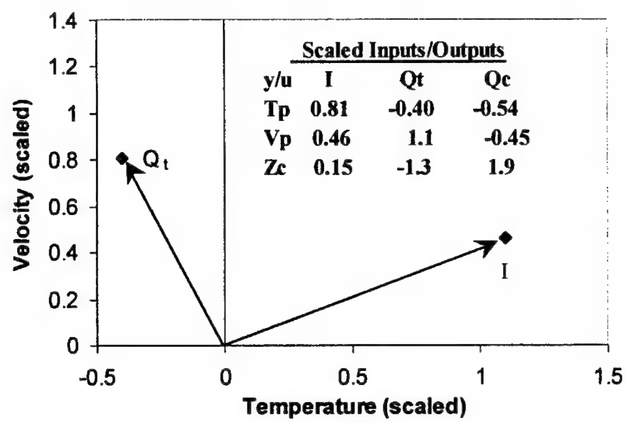

Figure 5: Scaled transfer matrix and plot of input/output interactions

These relationships can be illustrated (Fig.5) in a plot of the column vectors of G in the output space (Gevelber 1999). Here, each vector reveals how the outputs are affected when a single input is varied. These plots suggest that choosing current and torch gas flow rate can allow one to independently control particle, temperature, and velocity. Examination of the larger matrix structure provides insight for independent control of all 3 outputs.

When a feedforward controller was implemented based on the very linear results, it failed. Additional steady-state experiments were conducted to determine the reason for this failure. Instead of a sequential sweep of a single input, we varied the input conditions in a non-sequential manner. Plotting the output results for all the nominal

input cases revealed (Fig.6) that it is not possible to return to a nominal particle state. Thus closed loop control system is required to ensure that the desired spray conditions are achieved every time the torch is turned on and off and/or changed.

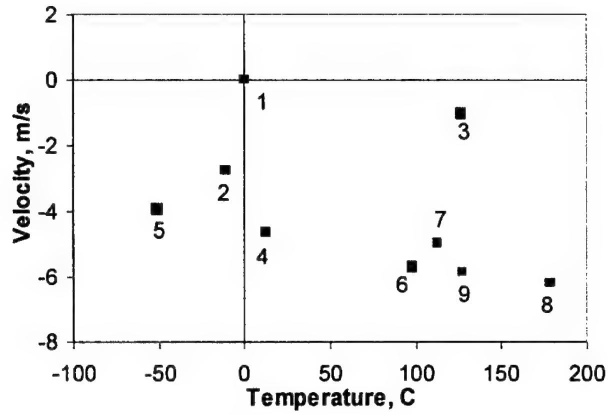

Figure 6: Variation in output state for repeated nominal input conditions

VI 2.1 Control Implementation

While there is now a variety of diagnostic equipment available, there has been no commercial closed loop particle-state control system on the market to date. One related strategy that is commercially available (such as from Praxair), is control of net plasma power. Fig 7 shows the capability that control of plasma enthalpy provides from our data set. While more data points are needed to be taken, it suggests that while control of plasma enthalpy has the right trends, it does not provide a tight control over particle temperature. Furthermore, net energy control does not ensure that particle velocity is controlled. Thus if one were to control the temperature by maintaining net torch power, one would expect that the velocity would float as a result.

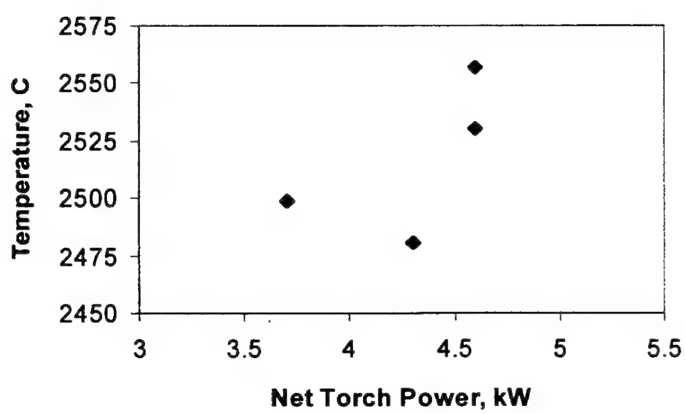

Figure 7: Particle temperature output for different plasma power inputs

Our initial closed loop design was to just control particle temperature by real-time feedback to current. A simple PID loop was tuned based on the experimental determined first order model, and the results to following a step command change of reference temperature is shown in Fig. 8.a. This loop was closed using the IPP sensor since its large measurement volume was insensitive to changes in centroid position. To simulate the affect of a disturbance, the torch gas level was changed by +/- 10% (Fig 8.b). These plots show that such as system will work. We are currently investigating what the performance limitations are as well as implementing control of simultaneous control of all 3 degrees of freedom (including centroid position).

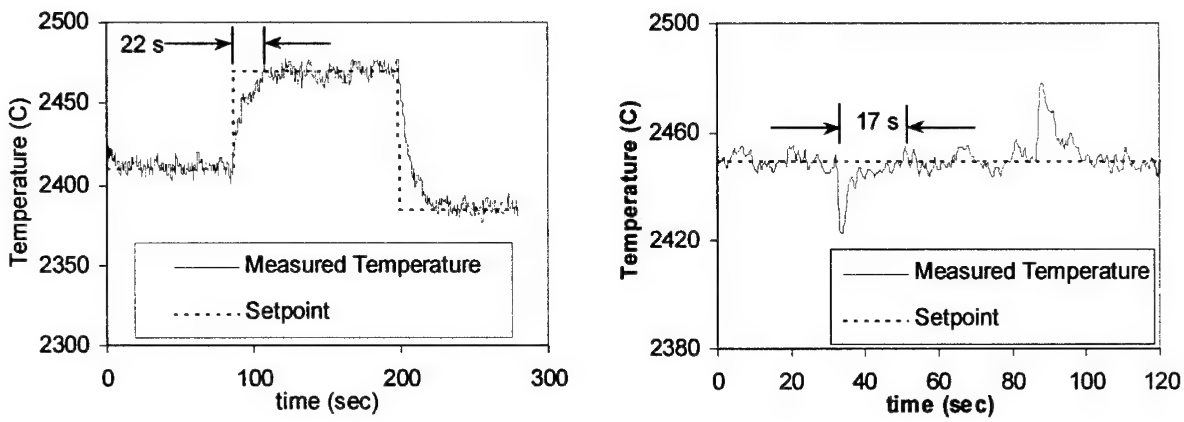

Figure 8: Closed loop control of temperature: (a) step response; (b) disturbance response

VI 2.2 Distribution Implications for Control Sensor Requirements

Feedback control approaches rely on robust, reliable sensors to provide an accurate measure of the process state. For plasma spray systems, the selection of suitable sensors will be dictated, to some degree, by the presence of distributions in particle state. As an example, consider the particle temperature measurements shown in Figure 9, obtained from the individual particle temperature and the IPP(a volumetric average based measurement). The individual particle temperature exhibits significantly higher temporal distributions compared to the IPP measurement, up to 2000 K variations.

Even when the signal is averaged over 1000 particles, the signal still displays fluctuations up to 60 K. This is on the same order as the control authority that can be achieved by variation of the torch inputs over reasonable ranges. If regulating average particle states is the main objective for control, then the IPP measurement may be more suitable for control since it rejects non-critical variability while preserving controllable fluctuations. If control of the distributions is desired, then the detailed information from the individual particle sensor would be required.

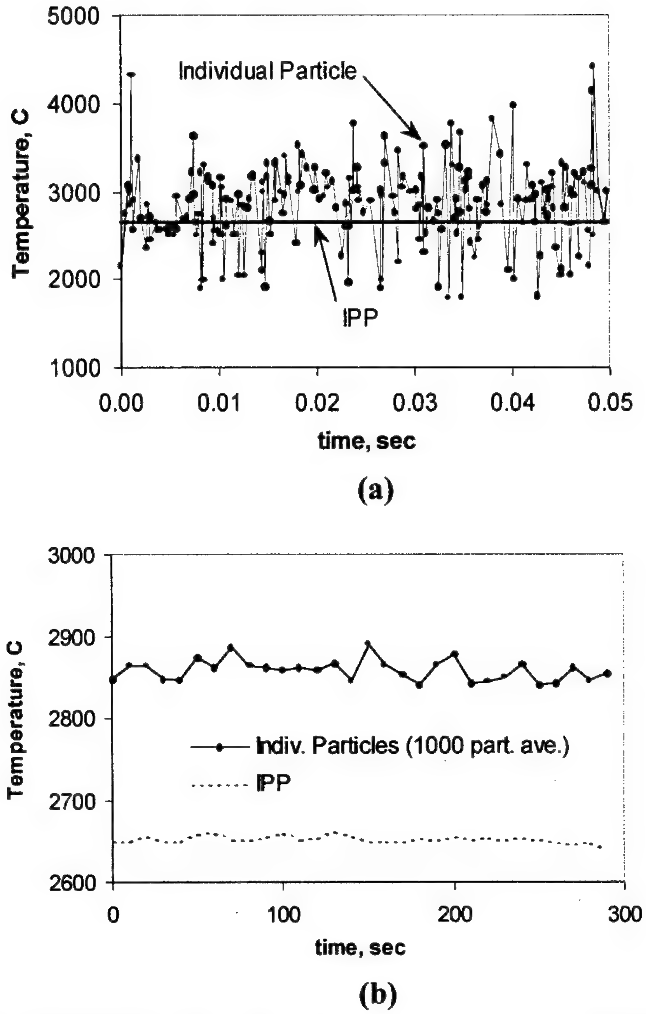

Figure 9: Comparison of individual particle temperature with IPP temperature: (a) un-averaged; (b) averaged.

VI.3 Advanced Czochralski Crystal Growth

The Czochralski crystal growth process has been one of the enablers of the microelectronic revolution. Process innovations that have expanded both the fundamental materials capabilities as well as improved production economics have been critical to enabling new applications in microprocessors, communications, and photonics as well as reducing cost that drive market penetration. The technical imperative for continued improvements is that substrate defects and structure significantly affect and/or limit the performance of the subsequent device layers. This is particularly true for emerging high performance and phontonic applications as well as sustaining further reduction in device size.

While the conventional approach to Czochralski system control has performed adequately to date for elemental semiconductors such as silicon (Si), it has not been explicitly designed to achieve the performance required for the increased electronic materials requirements for ULSI devices, overcome the performance problems being experienced in achieving scale-up to 300-400 mm boule diameter in Si, nor those problems encountered in achieving desired properties for

compound semiconductors for advanced opto-electronic applications such as gallium arsenide (GaAs), indium phosphide (InP), and Gallium Antimonide (which is difficult to grow but offers excellent performance for IR and microwave applications).

Advanced designs for future opto-electronic devices require the ability to engineer the materials properties of substrate materials in terms of dopant and electrically active defect distributions as well as control crystal shape. The conventional Czochralski control system design approach has two limitations. **First, the coupled nature of the process physics that can limit the achievable materials properties has not been explicitly considered in designing the control structure.** As such, closed loop control is used to maintain diameter, but there is no explicit coordination of control to achieve the ensemble of objectives. Thus, it is necessary that a richer set of control objectives be defined and implemented. **Second, the conventional control system designs do not directly address important process dynamics**, such as the time variation of the process, batch related disturbances, and the inherent performance limitations posed by some process dynamics and measurements. These problems have become particularly evident in trying to achieve the next generation of 300-400 mm boules.

A major foundation for the proposed work is the many previous studies that have analyzed the important process features such as the melt's thermal and fluid dynamics (Crochet), interface shape (Wilcox, Ramachandran), dislocation formation (Jordan, Motakef), defect formation (Voronkov, Brown, Sinno, Ammon), dynamics of the coupled melt, interface, and crystal (Derby, Hurle 1993) and diameter control system (Hurle 1977, Wilde). **These studies, however, do not provide a complete basis for system and control structure design.** In particular, the previous analyses do not provide an explicit representation of the system's input/output characteristics, dynamic features, or disturbances. Thus, another important foundation for the proposed research is our past work that developed a controls oriented understanding of the process, developing the knowledge base required to develop such an advanced control architecture (Gevelber 1987, 1988, 1993a,b, 1994a,b, Chen).

A primary tool developed has been the low order models that reveal the dominant time scales, dynamic features that pose fundamental limitations to achievable control performance, an cross-coupling between the multiple inputs and outputs that must be considered in developing such a coordinated control system. Recently, we have identified and explained the fundamental dynamics that determine process stability and the dynamic characteristics, and are thus able to relate choice of operating conditions that determine the systems eigenstructure (Chen). The new experimental puller funded through the DURIP grant has laid the ground work for determining how real puller system architecture and design choices affect the process and provide greater leverage over controlling a larger set of coupled objectives. In particular, we have been able to obtain real data sets of actuator levels and some states for several critical portions of the growth process.

VI 3.1 Advanced control requirements: competitive advantage through new materials capabilities and production improvements

We focus on two areas for improving processing capabilities: a) increasing the ability to manufacture material with new materials properties that enable new applications and/or extend the capabilities for existing applications, and b) improving the production objectives such as yield, rate, which reduces industries production costs.

A major need for an advanced control design stems from the batch nature of the process, i.e. the transformation of liquid to solid phase results in a decreasing melt level. The changing melt level results in both a changing thermal environment seen by the crystal as well as heat transfer throughout the melt and system. The change in heat transfer has two related impacts illustrated in figs.10: a) the energy balance about the interface changes, requiring a coordinated change of heater temperature and/or pull rate, and b) the dynamic eigenstructure of the process changes, requiring changing control gains.

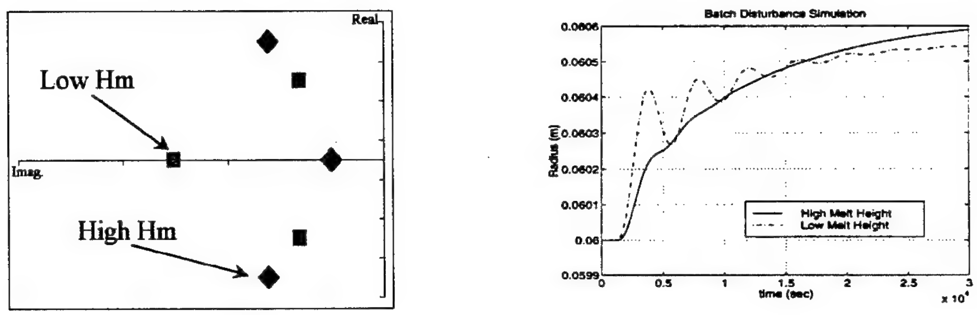

Fig.10 Change of eigenstructure and dynamic step response for different melt height levels

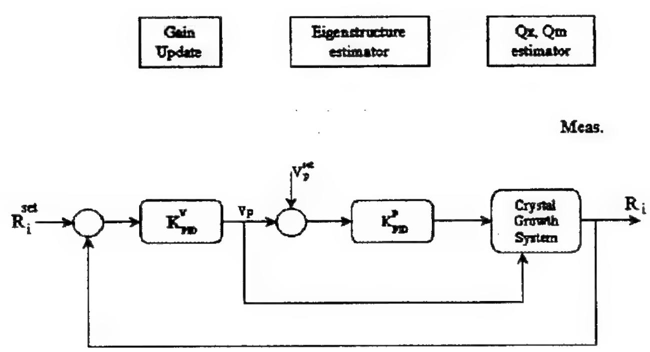

Fig.11
Conventional
(solid) and
proposed adaptive
(dashed) diameter

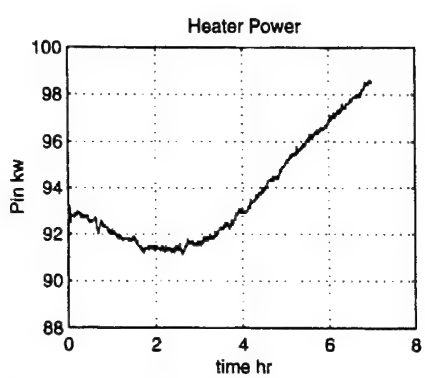

Fig.12 Heater power from a Kayex CG 6000 growth run (body section)

The current solution to compensate for both of these affects has been to implement a cascaded feedback structure (fig.11) that is gain scheduled. The outer loop of the cascaded control structure adjusts the pull rate to maintain a desired diameter, while the inner loop adjusts the heater power to maintain a desired pull rate. What makes this feedback approach difficult to implement is that the heater trajectory is parabolic (fig.12), requiring a sign change in controller gain. To date, when the sign change occurs is empirically determined. In addition, gain scheduling of the controller gains is required to compensate for the changing eigenstructure.

The difficulty of the current practice is that the tuning of the gain schedule is determined empirically, and is labor intensive since a whole growth run takes 24-48 hours to run. Since one is tuning the entire trajectory, this typically requires 2-4 runs to achieve the desired performance. Thus, one typically does not readily change process recipes. In addition, there is no automatic adaptation to system variations that occur as the heater package (insulation, graphite, and power supply) ages. Our analysis of both the control algorithm used (PII) and actuator records suggest that such a method requires large control gains to cancel the disturbance ramp input, which might have adverse impact on crystal quality.

Plots (fig.13) of the actuator trajectories (power and pull rate) and resulting crystal diameter during body growth, reveal several important issues for the conventional process. First is the large fluctuations in pull rate (+/- 10% of nominal), indicating use of large control gains, possibly inducing segregation (dopant) problems. Secondly, there is a large decrease in pull rate—reducing productivity by a factor of two. In the tail section (fig.14) in contrast, the system is under manual control. This results in a conservative operation, larger heater excursions, and entailing a long time period (reducing productivity).

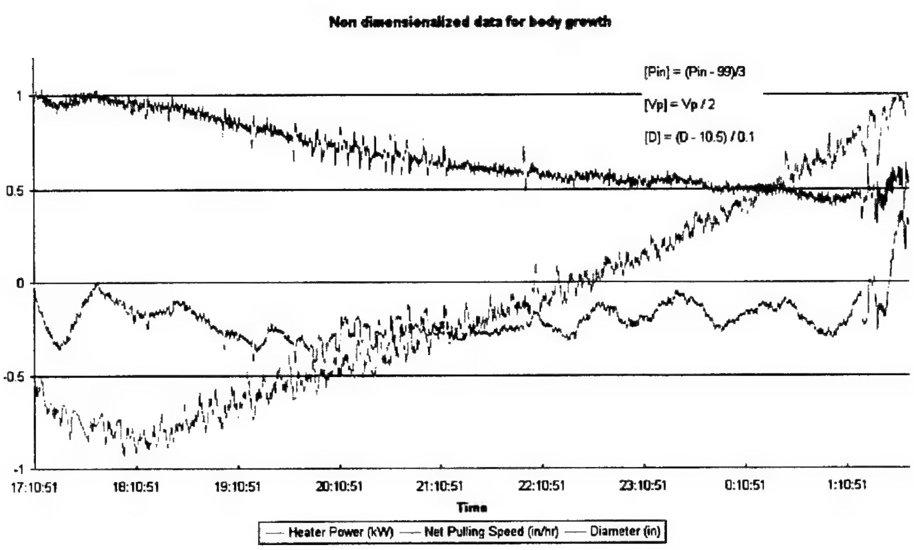

Fig.13 Actuator trajectories for body growth

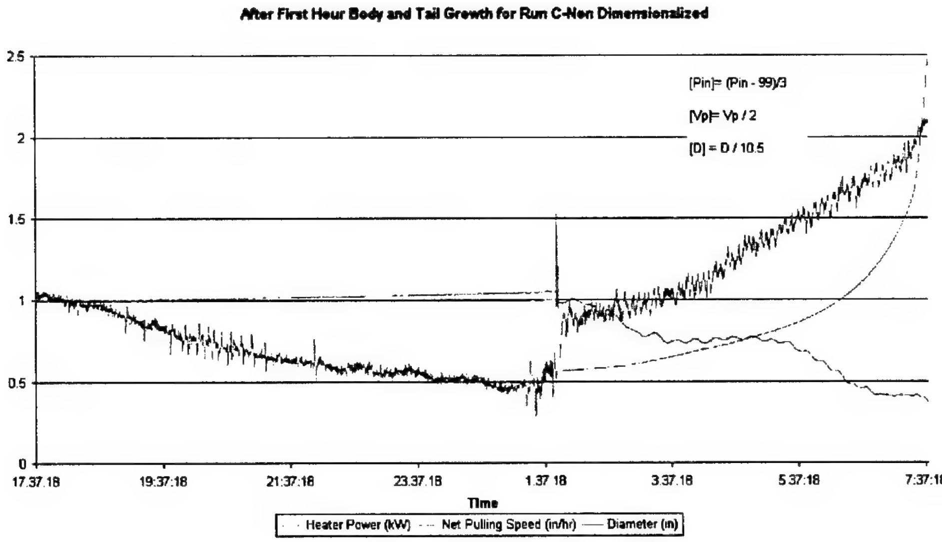

Fig.14 Actuator trajectories for body and tail growth

To compensate for these problems, we are proposing a two part solution: a) a measurement-based estimator/feedforward control scheme to compensate for the primary affects of the batch disturbance, and b) adaptive tuning scheme for the feedback controller to automatically update the required control gains. The feedback systems acts as a local trim to the error remaining after feedforward action is taken. Utilizing an adaptive scheme eliminates the need to manually determine the gain scheduling of the controller gains.

Discussions with both equipment vendors as well as commercial and research crystal growers have given us new insight into the need for specific advanced control requirements. An important part of this assessment was achieved through analysis of data sets for actual growth runs in commercial and experimental puller system. Analysis of these data sets reveal control requirements and complexities that had not been observed from analysis of "idealized" process models (both low order and high order methods). In addition, we seek to develop generic control methods that can be applied to a variety of different systems including both elemental and compound growth for a large variety of systems (e.g. liquid encapsulation, magnetic fields, heat shields, etc).

VI 3.2 sensor/estimation isssues

From a control point of view, one of the challenging issues will be in developing methods that can be used to control the tailing process. Minimizing tail length is an important productivity enhancer since one continues to pull at roughly the same very slow rate for a part of the crystal that can not be used commercially. As identified earlier by Gevelber, the weight measurement is subject to a right-half-plane zero. Analysis of achievable performance utilizing the Bode integral reveals that an output with a RHP zero poses a fundamental limitation to achievable performance. In practical terms, this limitation stems from the fact that the RHP zero limits the maximum gain in order to maintain stability since it is a non-minimum phase component. This gain limitation, can then be seen to limit how small one can make the error. These limitations have been experience by crystal growers who sought to tune their loops manually without the insight of there being a RHP zero, resulting in highly oscillatory and at times unstable control algorithms.

In terms of tailing, experimental results from growth runs at Kayex reveals some of the aspects of this problem. Fig.13 , shows a plot of both the actual diameter (obtained post growth) compared to the diameter estimate obtained form measurement the crystal weight. As the crystal initially begins to taper, the weight estimate captures the correct behavior. However, towards the end of the growth, an "anomalous" transient is observed wherein the crystal is still slowly decreasing while the weight signal reveals a sharp discontinuity, followed by a response that has the opposite sign of the real crystal shape. **The significance of this is that it would cause the closed loop controller to become unstable!** Potential loss of the entire crystal in order to implement a better tailing control would preclude such experiments from being considered. However, based on our improved understanding of the process dynamics, it is believed that we have developed a robust model of where the RHP dynamics originate from. While it is clear that from a closed loop control point of view, the closed loop performance would be limited in using weight as a feedback signal, we will analyze to what extent does such dynamics preclude a robust reconstruction/estimation based on multiple measurements.

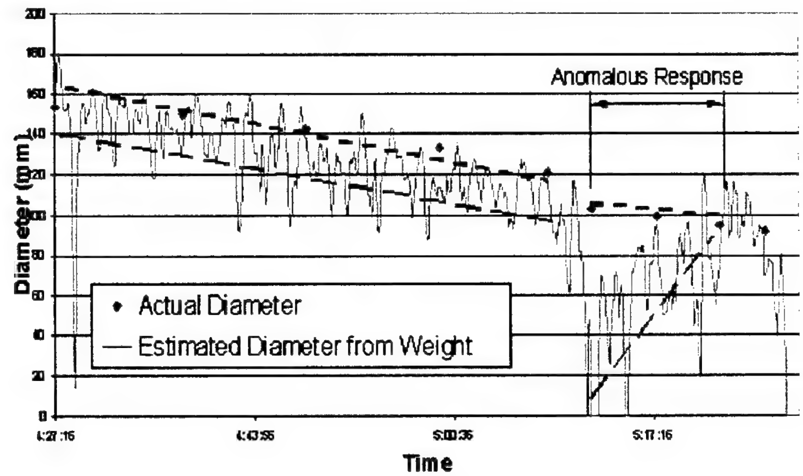

Fig.15 Comparison of actual diameter and estimated diameter from weight signal

VI.4 Real-Time Control of CVD

In many fields like tribology, corrosion, and optics, it is the surface properties of materials that govern their performance. Chemical Vapor Deposition (CVD) by virtue of being able to custom tailor the surface properties, has become an enabling technology for many critical applications in aerospace, engines, manufacturing, and microelectronics. Some of the important properties that can be engineered by design of the coating morphology and composition include thermal shock, corrosion, wear, and oxidation resistance. However, as application requirements become increasingly demanding, the ability to utilize a monolithic coating is limited, requiring the development of novel engineered structures involving multi-layers and multiphases.

An impediment to utilizing such advanced structures is the difficulty in determining the process recipe. In part this is due to the tighter control of morphology and composition required for these new coatings, which is difficult to achieve since growth conditions are still primarily determined by a combination of previous experience and empiricism.

Development of a controller based on in-situ measurements of the actual coating growth process is proposed as an aid to achieving higher yields, reducing time to market, aid in scaling up recipe to production scale processes, and providing new processing capabilities

Currently, CVD processes are essentially run in a feedforward manner where feedback is used only to maintain specific values of inputs such as flow rates and pressure and no feedback of in-situ measurements of the growth process is used(solid lines in fig16). Even in the well developed area of CVD for micro-electronic applications, we are aware of only several attempts to directly control the growth process --and this is limited to coating thickness(Gaffney,1995,Warnick). Thus, the selection of processing parameters (i.e. input settings such as gas flow rates and temperatures) to achieve materials objectives (e.g. composition and morphology), and processing objectives (e.g. layer thickness, growth rate, yield, and reproducibility) are developed based on previous processing knowledge and empirical experiments. The difficulty in using such an approach in developing new coatings is the inherent complexity of the process, in part due to having reaction paths where important constants, such as the kinetic coefficients, are not known. A feedback system can provide the capability to control coating microstructure in spite of these unknown parameters by utilizing real-time measurement of the growth process as the coating evolves.

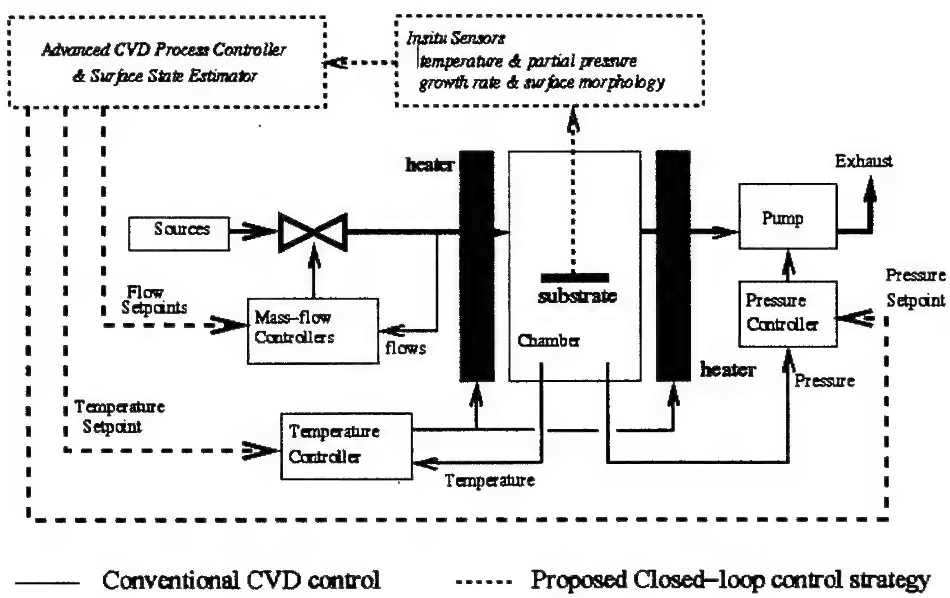

fig16. Controllers of CVD

Development of such a control system requires an explicit understanding of the process dynamics and input/output coupling to develop an appropriate control structure as well as development of advanced measurement/estimation capability to infer the local surface state. Our recent research results in terms of modeling and experimental analysis of CVD process dynamics (see VI 4.1) provide the knowledge base for this work. While

addressing new issues, our work has been based on the previous research work in terms of experimental investigations, modeling of the thermal/fluid/chemical nature of the process, fundamental nucleation and growth studies, and development of sensors to monitor the growth process. However no previous research, to our knowledge, has specifically focused on the critical issues needed to develop a real-time control of microstructure. Specifically, the majority of modeling studies of CVD, have focused on the steady-state characteristics, and have not identified the dynamic characteristics nor analyzed the multivariable aspects that are critical for control of morphology. We believe our results in this area are new and provide the required basis to achieving closed loop control of coating growth. Additionally, while there have been separate studies of the thermal/fluid nature of the growth process and fundamental work on nucleation and growth modeling, only limited research, primarily empirical investigation of specific systems, have attempted to combine the two areas. Our experimental work indicates that our approach is valid.

In developing a closed loop control system, it is important to consider the different time scales that are relevant. Time scales related to disturbances and command signals indicate the required bandwidth of the closed loop system. If the growth process kinetics vary with coating thickness, this may result in a ramp disturbance over T_{batch}. Consideration of controlling dominant features, such as grain size, suggest a feature time period related to the feature size. In contrast, the physical time constants have been shown for our reactor to be on the order of $T_{physics} \sim 5$ - 30 seconds. Thus, for slow growth rates, it would be appropriate to design a closed loop control using slow time constants, (i.e. longer than $T_{physics}$ and T_{delay}), and one should be able to achieve the desired control performance without the limitations posed by the RHP zero dynamics and the transport delay, T_{delay}. However failure to consider these dynamic limitations in setting the closed-loop bandwidth (i.e. by setting the control gain too high), could result in closed-loop instability. Operating the system in this condition can be thought of as controlling the system in a quasi-steady-state. Under the faster growth conditions, however, one must take into consideration the dynamics of the process by developing an appropriate control algorithm.

A parametric analysis of the eigenstructure and how it varies (Toledo Quinones,1996) indicates that the dominant time constants are those associated with the bulk reactor flow dynamics (i.e. the surface reactions are significantly faster). Consideration of the bulk flow dynamics reveal that $\tau_{flow} \sim \forall$ Q (i.e. the time constant scales with the reactor volume divided by flow rate). Thus, even for the growth rates reported above, where $T_{feature} >> T_{physics}$ as the reactor is scaled up, this will no longer hold true. Since in many industrial applications a batch process is used to coat multiple parts, it is likely that in production scale reactors, dynamics must be considered for developing effective closed-loop controllers.

To gain insight into important dynamic features and relative performance of actuators, we linearize the nonlinear model is linearized at various operating points The validity of

using a linear analysis is confirmed by a comparison of the linear and nonlinear model predictions(Gevelber 1998). Frequency analysis provides important insight into each inputs gain (magnitude impact of changing an input) as well as bandwidth (how fast it acts). The equations are non-dimensionally scaled to represents the fractional change of deposition rate for a fractional step increase for each of the inputs in order to compare the performance of inputs with different units. (see Gevelber 1995 for more details)

At higher temperatures, the frequency analysis reveals that the total flow rate, T_iCl_4 mass flow rate, and pressure have the largest gain, followed by heater power and mass flow rates for N_2 and H_2. This is observed in the dynamic simulations of a step response to a 2% change of each input. Pressure and heater power inputs, however, have a greater bandwidth by an order of magnitude.

In this regime, a right-half-plane (RHP) zero is present for the pressure input, which would limit the achievable closed loop performance. The transient signature of a RHP zero can be observed (the initial response is in the opposite direction of the steady-state) for P_{chmb}. The RHP zeros are present due to the relative dynamics of $\bar{Q}$ and $P_{T_iCl_4}$ as described in our paper. The importance of the RHP zero is that it limits the achievable control performance. The transport delay also introduces a right-half-plane zero for variations in reactant rate as shown in.

VI 4.1 experimental results and analysis

To confirm our dynamic model and to investigate proposed control designs, we have constructed an experimental CVD unit (fig.2). Primary instrumentation includes the use of a microbalance to obtain real-time measurement of deposition rate, thermocouple arrays, and real-time mass spec for partial pressures. Fig.18 shows the good agreement between our model predictions of partial pressure dynamics within the reactor for a change in mass inlet flow and the experimentally measured values. Important characteristics confirmed include the mass transport delay and defective RHP zero transient. These factors are significant since they pose a fundamental limitation and must be taken into account when designing the control algorithm. We have also discovered the significant difference in open-loop dynamics between use of a throttle valve used before the vacuum pump (where $\tau \sim \forall C_v$, which is slow) and a by-pass bleed of N_2 (where $\tau \sim \forall Q_{pump}$, which is fast). Confirmation of these results is shown in fig.17. A more significant test of our model predictions is the experimental confirmation of our claim that while closed loop pressure control yields fast total pressure responses, the partial pressure scale as $\tau \sim \forall Q_{exhaust}$.

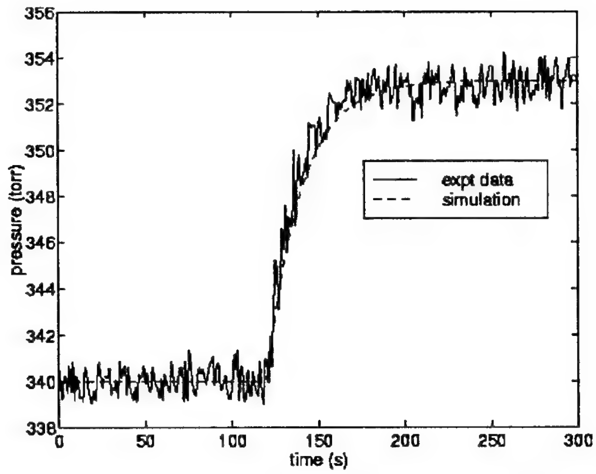

fig 17. Open loop pressure response: comparison of experimental data and model prediction

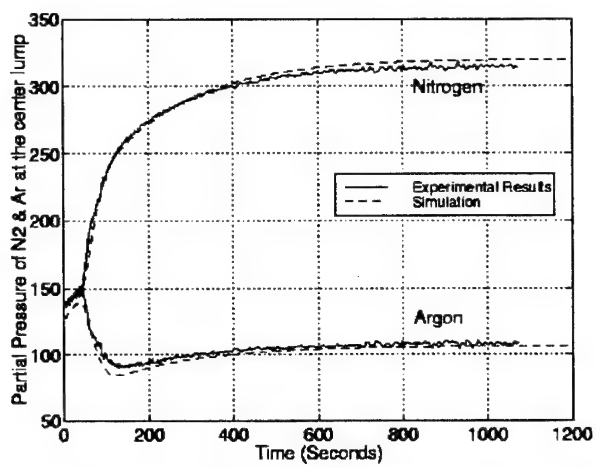

fig 18. Open loop response to increase in N_2 flow: comparison of partial pressure measurements to model

VII Industrial Collaboration/Technology Transfer

For each of the projects, we have established collaborative relationships with both equipment vendors and end-users of these processes, as well as tie-ins to Air Force/Government Laboratories to insure relevance. Some of these relationships are more formal where the companies are interested in sharing research results, where others are more informal consulting type relations.

Plasma Deposition: Thermal spray is widely used for manufacture of coatings due the ability to engineer unique ceramic/metallic coatings at high rates and low cost. Widely used for turbine and engine components, it also has promise for being able to manufacture coatings in new advanced components such as fuel cells and space propulsion systems.

We have developed contacts with some of the traditional application areas such as

turbines/engines including Y-C Lau at GE's CRD, Steve Hay at UTRC as well as the manufacturing operation at Pratt & Whitney's North Berwick plant (UTC). Discussions with one equipment vendor (Crawmer at Praxair) has made us aware of the potential new market opportunities that will exist once better control capability is in place. These application areas include semiconductors, biomedical, and nanophase particles. While nanophase particles offer the potential to yield new material properties for such coatings, Monica Stucke's investigation at WPAFB had difficulties due to reproducibility problems, a perfect opportunity for improved control.

Other application areas include coatings for space propulsion systems. Dave Ellis of NASA Glenn has developed a new plasma sprayed coating for the combustion chamber. In this application, a material that is strong, yet able to take a large temperature gradient and conduct the heat away is required. The current method is to centrifugally cast the component, so a plasma sprayed manufacturing processes would be significantly cheaper. Richard Homes of NASA Marshall indicates that there will possibly be the need to achieve better control the coating characteristics once the core technology has been demonstrated. The novelty of the proposed coating is that it is a copper matrix intermetallic, with an overspray of NiCr to prevent oxidation.

Besides Praxair, we are also connected to TAFA of New Hampshire. Vladimir Belaschenko, VP of RD, TAFA has discussed with us the importance of developing a better interpretation of sensor capability that they currently market. Most spray users do not have the capability to interpret what these spray pattern temperature/velocity sensors are telling them, nor is the knowledge base available to utilize them for real time control. Along these lines, we have started a collaboration with Dr. James Fincke of INEEL to conduct a series of experiments that map out the need for control and interpretation of some of the basic sensors that are available that are based on ensemble measurements.

Crystal Growth: While there are important similarities in approaching the control problem for Czochralski systems, each material system has particular issues that must be addressed. In particular, issues for Silicon are focused on problems associated with scale up of the process to 300-400 mm, whereas compound semiconductors such as InP and GaAs are focused both on improving the fundamental materials capabilities as well as improving yield. In addition, new Air Force missions in space, particularly satellite arrays that require high bandwidth for communications as well as detector arrays for missile launches pose important drivers for developing new crystal substrates. Besides the need for high yield InP and GaAs, better control of the bulk crystal growth process is needed for other difficult to grow systems suchas Gallium Antimonide. This material offers excellent performance for IR and microwave applications, but has defect problem during manufacture that limit its application.

For compound semiconductors, we focus on two main efforts: InP and GaAs. For InP, our primary collaborator is David Bliss's research/process development effort at AFRL/SNHX (Hanscom AFB). A new puller system design has been developed for AFRL by GTI, and M/A will be the operator who will be growing the initial crystals. We

have established collaborative relations with all three of these partners (Dr. Gupta at GTi and Drs. Doug Carlson and Roland Ware of M/A COM). Critical issues for InP are improving yield, which is primarily limited due to twinning. Following D. Hurle's recent analysis, twinning can be attributed to growth variations through a critical angle. Thus achieving good diameter control during growth is critical. The difficulty, however, is achieving this under low gradients which is desirable to minimize dislocation density. Low gradients, however, tend to make the system more sensitive to system perturbations. Similarly, we are also discussing GaAs requirements with M/A COM who are a major manufacturer of SI GaAs used in DOD and civilian applications. At the recent ICCG-13, we have also established contact with the major InP and GaAs research efforts in Germany and France. Development of good diameter control under low gradients continues to be a critical problem, and shares to some extent some of the adaptive control requirements for silicon described below. Note, the proposed Varian system can be modified to liquid encapsulated (LEC) which is used for compound systems.

In the area of silicon, there are two major focuses: those issues arising from the scale up to 300-400 mm, and issues related to improving yield and time to market for conventional scale pullers. Our primary contact from equipment vendors is Kayex, a division of General Signal in Rochester, while our primary end-user is MEMC. (We have however meet with representatives from Komatsu and Wacker.) One area that overlaps with compound systems is the need to develop adaptive control for the body growth. Currently, the diameter control gains are time-varying, but must be tuned by hand. This is very time consuming since a number of full batch processes must be done, each taking 1-2 days. Control of the neck is also critical, especially for scale up to 300 mm since the neck will now support a large weight. Lastly, for large scale systems, users are finding loss of control authority from the heaters due to the thermal lag of the system. Our melt control strategy is expected to solve this problem.

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